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(54) Title: VODKA AND A PROCESS FOR THE PRODUCTION OF VODKA

(57) Abstract: An improved process is provided to prepare vodka with excellent properties comprising mixing water and absolute alcohol, treating the mixture with activated coal followed by filtration, adding sugar, aroma compounds and optionally other ingredients characterized in that the mixture of water and alcohol after the treatment with activated coal is cooled down to a temperature of about - 10° C to 15° C, at which temperature the mixture is maintained for about 4 - 8 hours, after which the resulting mixture is filtered, gradually adapted to room temperature, aroma and optionally other ingredients are added to the filtrate and optionally the resulting mixture at room temperature is filtered again before bottling. A new vodka is provided comprising a percentage of absolute alcohol in water of about 35 - 50 vol %, 4 - 6 mMol/l sugar, 0.05 - 0.2 mMol/l of sodium bicarbonate, and 0.02 - 0.04 vol % of extract of flax seeds of first discharge.

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Vodka and a process for the production of vodka

The present invention relates to vodka and to a process to prepare vodka comprising mixing water and absolute alcohol, treating the mixture with activated coal followed by filtration, adding sugar, aroma compounds and optionally other ingredients.

At present, a widely known method of production of vodka is consecutive mixing of water-alcohol liquid with sugar syrup, various aroma compounds like for instance synthetic aromatizers for disguising the fusel odor of alcohol, optionally other ingredients and subsequent filtering of the mixture [*Retseptury likerno-vodochnykh izdelii i vodok* (Formulas of alcoholic beverages and vodkas) Moscow: Legkaya promyshlennost, 1981].

Such method is rather simple technologically, however, the vodka produced by this method has insufficiently high organoleptic parameters. Sugar settles on the surface of activated coal decreasing its activity and adsorbability. It decreases the filtration level of the mixture and the ready vodka manifests the residual traces of fusel oils. In addition, the vodka produced by the known method has a strong odor of the introduced aromatizer and the beverage has an unnatural "synthetic" odor. The aromatizer evaporates with time and, accordingly, alcoholic aftertaste increases in the odor and taste. In addition, such vodka has usual ordinary taste and insufficiently high organoleptic parameters.

Russian patent no. 2171283 discloses a method of production of vodka by obtaining water-alcohol liquid, filtration, mixing of the water-alcohol liquid with sugar syrup and other ingredients. According to this method the

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obtained water-alcohol liquid is filtered in a column with activated coal, then sugar and other ingredients of the formula of vodka are introduced in the obtained purified water-alcohol liquid, it is mixed, kept until it becomes homogeneous, and filtered prior to bottling (Russian Patent 2171281).

Organoleptic parameters of this vodka are better than in the vodka produced by the method first mentioned. However, fusel oils present in the water-alcohol liquid still form a film on the surface of activated coal and this retards the technological process, as the flow rate through the column is slowing down. The lower filtration rate impairs, accordingly, the taste of vodka.

It is an object of the present invention to provide a process which makes it possible to improve organoleptic parameters of vodka.

The present invention provides a process as mentioned in the preamble characterized in that the mixture of water and alcohol after the treatment with activated coal is cooled down to a temperature of about - 10 °C to - 15 °C, at which temperature the mixture is maintained for about 4 - 8 hours, after which the resulting mixture is filtered, gradually adapted to room temperature, aroma and optionally other ingredients are added to the filtrate and optionally the resulting mixture at room temperature is filtered again before bottling. Normally room temperature will be in the range of about 18 °C - 25 °C.

Preferably, the filtrate is adapted to room temperature by pumping the filtrate to a non-isolated tank until room temperature has been attained.

More preferably, the aroma compounds applied in said process comprise extract of flax seeds.

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Still more preferred, water with an alkalinity of less than 3 meq/l is applied for the process of the present invention.

In a preferred embodiment of the process of the invention the mixture after cooling down is filtered through a carbon filter, preferably a Z-carbon filter.

In still a preferred embodiment of the process of the invention the resulting mixture at room temperature is filtered over a series of micro filters before bottling, preferably immediately before bottling.

Preferably, water for the water-alcohol liquid is obtained by mixing softened water and water treated by the method of reverse osmosis, normalized by the alkalinity parameter within about 2-3 meq/l.

Furthermore, the water-alcohol liquid is preferably treated with activated coal in the coal column at a filtration rate of 40-50 decaliters/hour.

The present invention further provides a vodka comprising

- a percentage of absolute alcohol in water of about 35 - 50 vol %,
- 4 - 6 mM sugar
- 0.05 - 0.2 mM of bicarbonate, preferably sodium bicarbonate and
- 0.02 - 0.04 vol % of extract of flax seeds of first discharge.

Preferably said vodka comprises a minor amount of impurities per 1 absolute alcohol of

- acetic aldehyde lower than 3 mg
- fusel oil lower than 6 mg
- ester lower than 5 mg
- methyl alcohol lower than 0.2 ml and

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with an alkalinity characteristic of less than 3 meq, more preferably less than 2 meq.

Throughout this patent application the wording  
5 alkalinity characteristic has been defined as volume in ml of hydrochloric acid with a concentration 0.1 M (viz. 0.1 Mol/l) of HCl used for titration of 100 ml of vodka.

Still more preferred the vodka of the present  
10 invention comprises

- a percentage of absolute alcohol in water of about 40 vol %,
- 5.3 mM of sugar,
- 0.12 mM of sodium bicarbonate and
- 15 - 0.032 vol % of extract of flax seeds.

The objects of the present invention are accomplished in the following way:

to prepare water for water-alcohol liquid the mixture  
20 is composed of softened water and water treated by the method of reverse osmosis, normalized by the parameter of alkalinity within 2-3 meq/l. Then the obtained water is mixed with ethyl alcohol, then the obtained water-alcohol liquid is passed through the coal column with activated  
25 coal at the filtration rate of 40-50 decaliters/hour. The treated mixture is directed to the heat isolated cooler for cooling to -10 °C - -15 °C, then it is pumped at this temperature to a heat isolated tank and kept there during 4-8 hours. After keeping, the cooled mixture is filtered  
30 through a Z-carbon filter and pumped to a non-heat isolated tank for "rest" until the room temperature of about 18 °C - 25 °C is gradually attained. Sugar syrup is added to the obtained water-alcohol liquid and if desired, other ingredients of the vodka to be prepared and for instance

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aromatic alcohol. Prior to bottling, the produced vodka is consecutively filtered through a series of microfilters.

Sugar can be added at any moment of the process, the other ingredients are preferably added after the mixture  
5 hab be brought to room temperature.

Profound cooling to the temperature of  $-10^{\circ}$  -  $-15^{\circ}\text{C}$  considerably increases the density of the water-alcohol liquid, and leads to formation on walls of the cooler of a fine crystalline film being a mixture of aldehydes, fusel  
10 oils, and methanol. Filtration at such low temperature still better purifies the water-alcohol liquid while not destroying the molecular system water-alcohol formed at cooling of the water-alcohol liquid. The same aim is attained by gradual natural warming of the mixture after  
15 filtration with an appropriate time lag.

The present invention will be exemplified further by the following example which is not to be considered as restricting the scope of the invention in any way.

#### Example

Vodka "Stolichnaya Elit" is prepared using alcohol "Lux" and water obtained by mixing softened water with  
25 water preliminarily treated by the method of reverse osmosis, normalized by a parameter of alkalinity within 2-3 meq/l. Alcohol or spirit "Lux" has been described in the State Standard of the Russian Federation,  
no. P 51652-2000.

30 With regards to its physical and chemical indicators spirit "lux" must conform the requirements in the table below.

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<u>Indicator</u>	<u>Norm for the spirit</u>
Volume of ethyl spirits, %, not less than	96,3
Sulphuric acid test for identification of purity	Passed
Oxidability test, min., 20 °C, not less than	22
Mass concentration of aldehydes to absolute alcohol, mg/dm <sup>3</sup> , no more than	2
Mass concentration of fusel oil: - 1 propanol, 2-propanol, isobutyl alcohol, 1-butanol, isoamyl alcohol to absolute alcohol, mg/dm <sup>3</sup> , no more than	6
Isoamyl and isobutyl alcohols (3:1) to absolute alcohol, mg/dm <sup>3</sup> , no more than	2
Mass concentration of ester to absolute alcohol, mg/ dm <sup>3</sup> , no more than	5
Volume of methyl spirit to absolute alcohol, %, no more than	0,03
Mass concentration of free acids (with no CO <sub>2</sub> ) to absolute alcohol, mg/dm <sup>3</sup> , no more than	8
Mass concentration of solid residual to absolute alcohol, mg/dm <sup>3</sup> , no more than	-
Mass concentration of basic volatile nitrogens to nitrogen, in 1 dm <sup>3</sup> of absolute alcohol, mg, no more than	-

Spirit "Lux" is produced from various sorts of grains and mix of grain and potatoes, volume of starch in the mix should not exceed 35% for production of spirit "Lux".

In a mixing tank the alcohol and water are mixed to obtain the mixture of 40%, then the mixture is passed through the column with activated coal at the filtration rate of 40-50 decaliters/hour. The filtered water-alcohol

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mixture is passed through the heat exchanger to decrease its temperature to  $-10^{\circ}\text{C}$  -  $-15^{\circ}\text{C}$ , the cooled mixture is kept in the heat isolated tank during 4-8 hours depending on the rate of formation of the crystalline film on walls of the tank. The cooled mixture is filtered through a Z-carbon filter, the temperature of the mixture is increased by  $6^{\circ}\text{C}$  -  $10^{\circ}\text{C}$ , the obtained filtrate is pumped to a non-isolated tank for "rest" until the room temperature of  $18^{\circ}\text{C}$  -  $25^{\circ}\text{C}$  is gradually attained. To the obtained water-alcohol liquid 65.8% sugar syrup is added and, other ingredients of the vodka and aromatic alcohol.

To the other ingredients belongs the use of grains for production of extract of flax for 1000 dal. of vodka as given in the following tabel:

Name of raw material	Use, in kilos	Liquid of water and spirit		Time of extraction, in days	Extract of the first discharge (filtered)		
		Volume in liters	Volume in %		Volume, in liters	Volume in %	% from water and spirits liquid
Seeds of flax	0.4	4.0	50	5	3.2	49	80

The obtained composition is kept for 1-5 hours and then the produced vodka is, prior to bottling, consecutively filtered through a series of micro filters and bottled.



The ready for use vodka has the following parameters:

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Strength (volume %)	40.0
Appearance	Transparent, colorless liquid with glitter
Taste	Soft, harmonious, with rounded vodka shades
Aroma	Characteristic of vodka, without admixture of fusel oil odor

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The produced vodka received 9.8 points from tasters.  
The following tabel gives more characteristics:

# **COMPOUNDING OF VODKA STOLICHNAYA ELIT**

## **Indices of Vodka**

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### **Physicochemical**

	Volume, %	40,0
10	Alkalinity- volume of hydrochloric acid with concentration with (HCL)=0,1 molecule/dm <sup>3</sup> , used for titration of 100 cm <sup>3</sup> of vodka, cm <sup>3</sup> , not exceeding	2,0
15	Mass concentration of acetic aldehyde in 1 dm <sup>3</sup> of absolute alcohol, mg, not exceeding	3,0
	Mass concentration of fusel oil (1-propanol, 2-propanol, iso-butanol, 1-butanol, iso-amylol) in 1dm <sup>3</sup> of absolute alcohol, mg, not exceeding	6,0
20	Mass concentration of ester, in 1 dm <sup>3</sup> of absolute alcohol, mg, not exceeding	5,0
25	Volumetric share of methyl alcohol, absolute alcohol equivalent, %, not exceeding	0,02

### **Organoleptic**

30	Appearance	clear liquid without extraneous impurities and sediment
	Color	colorless
	Taste	soft, peculiar to vodka
	Aroma	specific vodka aroma

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### **MIXING FOR 1000 DAL**

	Name of component	Measuring unit	Quantity
40	Ethyl spirit rectified "Lux"	L	Spirit and water equivalent to
	Potable water improved	L	volume of 40%
	Sugar syrup, 68%	L	20,7
45	Extract of flax seeds of 1 <sup>st</sup> discharge	L	3,2
	SodiumBicarbonate	kg	0,1

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**CONSUMPTION OF INGREDIENTS FOR 1000 DAL OF VODKA**

<b>Name of Ingredients</b>	<b>Quantity (kg)</b>
Sugar granulated purified	18,0
Sodium bicarbonate	0,1
Oil Flax (seeds)	0,4

10        The present invention provides a process which makes  
it possible to produce vodka with improved organoleptic  
parameters.

**Claims**

1. Vodka, comprising

- a percentage of absolute alcohol in water of about 35 - 50 vol %,
- 4 - 6 mM sugar
- 0.05 - 0.2 mM of bicarbonate, preferably sodium bicarbonate
- 0.02 - 0.04 vol % of extract of flax seeds of first discharge.

2. Vodka according to claim 1 with an amount of impurities / 1 absolute alcohol of

- acetic aldehyde lower than 3 mg
- fusel oil lower than 6 mg
- ester lower than 5 mg
- methyl alcohol lower than 0.2 ml and with an alkalinity characteristic of less than 3 meq.

3. Vodka according to claim 1 or 2 comprising

- a percentage of absolute alcohol in water of about 40 vol %,
- 5.3 mM of sugar,
- 0.12 mM of sodium bicarbonate and
- 0.032 vol % of extract of flax seeds.

4. Process to prepare vodka comprising mixing water and absolute alcohol, treating the mixture with activated coal followed by filtration, adding sugar, aroma compounds and optionally other ingredients characterized in that the mixture of water and alcohol after the treatment with activated coal is cooled down to a temperature of about - 10 °C to - 15 °C, at which temperature the mixture is

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maintained for about 4 - 8 hours, after which the resulting mixture is filtered, gradually adapted to room temperature, aroma and optionally other ingredients are added to the filtrate and optionally the resulting mixture at room  
5 temperature is filtered again before bottling.

5. Process to prepare vodka according to claim 4 whereby the filtrate is adapted to room temperature by pumping the filtrate to a non-isolated tank until room temperature has  
10 been attained.

6. Process to prepare vodka according to claim 4 or 5 whereby the aroma compounds comprise extract of flax seeds.

15 7. Process to prepare vodka according to claim anyone of the claims 4 - 6 whereby water with an alkalinity of less than 3 meq/l is applied.

20 8. Process to prepare vodka according to anyone of the claims 4 - 7 whereby the cooled down mixture is filtered through a carbon filter.

25 9. Process to prepare vodka according to anyone of the claims 4 - 8 whereby the resulting mixture at room temperature is filtered over a series of micro filters before bottling.

# INTERNATIONAL SEARCH REPORT

PCT/NL 03/00676

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C12G3/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C12G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ, FSTA, BIOSIS, COMPENDEX

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Section Ch, Week 200143 Derwent Publications Ltd., London, GB; Class D16, AN 2001-406599 XP002264203 & RU 2 167 932 C (BRYANSKSPIRTPROM STOCK CO), 27 May 2001 (2001-05-27) abstract	1-3
A	---	4-9
X	DATABASE WPI Section Ch, Week 200203 Derwent Publications Ltd., London, GB; Class D16, AN 2002-024524 XP002264985 & RU 2 175 010 C (BOBRYSEV S YU), 20 October 2001 (2001-10-20) abstract	1-3
A	---	4-9
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Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents:

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# INTERNATIONAL SEARCH REPORT

PCT/NL 03/00676

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	<p>DATABASE WPI  Section Ch, Week 200051  Derwent Publications Ltd., London, GB;  Class D16, AN 2000-557186  XP002264205  &amp; RU 2 144 949 C (VLADALCO STOCK CO),  27 January 2000 (2000-01-27)  abstract</p> <p>---</p>	1,3
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X	<p>DATABASE WPI  Section Ch, Week 199622  Derwent Publications Ltd., London, GB;  Class D16, AN 1996-220420  XP002264207  &amp; RU 2 044 045 C (MOSC KRISTALL WKS STOCK  CO), 20 September 1995 (1995-09-20)  abstract</p> <p>---</p>	4-9
A	<p>DATABASE WPI  Section Ch, Week 200013  Derwent Publications Ltd., London, GB;  Class D16, AN 2000-145962  XP002264208  &amp; RU 2 120 965 C (KOSITSKII E YA),  27 October 1998 (1998-10-27)  abstract</p> <p>-----</p>	1-9

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Information on patent family members

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